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Hardmetals — Metallographic determination of microstructure —

Part 3:
Measurement of microstructural features in Ti (C, N) and WC/cubic carbide based hardmetals

Métaux-durs — Détermination métallographique de la microstructure —

Partie 3: Mesure des caractéristiques des microstructures des métaux-durs à base de carbures Ti (C, N) et WC/cubiques

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

ISO 4499 consists of the following parts, under the general title *Hardmetals — Metallographic determination of microstructure*:

— *Part 1: Photomicrographs and description*

— *Part 2: Measurement of WC grain size*

— *Part 3: Measurement of microstructural features in Ti(C,N) and WC/cubic carbide based hardmetals*

— *Part 4: Characterisation of porosity, carbon defects and eta-phase content*

Introduction

This part of ISO 4499 essentially covers the following topics:

- materials types and phases to be measured including the following:
    - Ti(C, N) cermets;
    - WC/Cubic carbide hardmetals;
- preparation methods to highlight differences between conventional WC/Co hardmetals and materials containing cubic phases;
- linear analysis techniques to acquire sufficient statistically meaningful data for phase quantification;
- analysis method to calculate representative average values;
- reporting to comply with modern quality requirements.

Hardmetals — Metallographic determination of microstructure —

Part 3:
Measurement of microstructural features in Ti (C, N) and WC/cubic carbide based hardmetals

1 Scope

This part of ISO 4499 gives guidelines for the measurement of microstructural features in Ti(C,N) based hardmetals and WC/Co hardmetals that contain additional cubic carbides by metallographic techniques only using optical or electron microscopy. It is intended for sintered hardmetals (also called cemented carbides or cermets) containing primarily inorganic carbides and nitrides as the hard phase. It is also intended for measuring the phase size and distribution by the linear intercept technique.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4499–1:2008, *Hardmetals — Metallographic determination of microstructure — Part 1: Photomicrographs and description*

ISO 4499–2:2008, *Hardmetals — Metallographic determination of microstructure — Part 2: Measurement of WC grain size*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
nano
with carbonitride or cubic carbide phase size <0,2 µm, respectively

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.2
ultrafine
with carbonitride or cubic carbide phase size 0,2 µm to 0,5 µm, respectively

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.3
submicron
with carbonitride or cubic carbide phase size 0,5 µm to 0,8 µm, respectively

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.4

fine

with carbonitride or cubic carbide phase size 0,8 µm to 1,3 µm, respectively

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.5

medium

with carbonitride or cubic carbide phase size 1,3 µm to 2,5 µm, respectively

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.6

coarse

with carbonitride or cubic carbide phase size 2,5 µm to 6,0 µm, respectively

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.7

extra coarse

with carbonitride or cubic carbide phase size >6,0 µm, respectively

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.8

Ti(C, N) cermets

TiCN-based cermet contains 3 to 30 weight % of a binder phase mainly composed of Co and/or Ni, but may also include Mo

Note 1 to entry: The balance being substantially a hard phase and a few minor impurities.

Note 2 to entry: The hard phase is mainly composed of titanium carbide, nitride and/or carbonitride, but may also include carbonitrides of (Ti,Ta), (Ti,W) or (Ti,Ta, W).

Note 3 to entry: These materials typically contain hard phases that can have grains with a core/rim structure.

3.9

WC/Cubic carbide hardmetals

hexagonal WC-based hardmetals containing substantial amounts of a carbide having a cubic lattice, such as, for example TiC or TaC, and which can contain W in solid solution

Note 1 to entry: These materials typically contain hard phases that may have grains with a core/rim structure.

Note 2 to entry: See Table 1.

3.10

phase region

single constituent of the hardmetal like WC, cubic carbide or binder

4 Symbols and units

A	area, in square millimetres (mm^2)
ECD	Equivalent Circle Diameter of a specified phase, in micrometres (µm)
L	total line length in a specified phase, in millimetres (mm)
l_i	measured length of individual intercepts in a specified phase, in micrometres (µm)
$\sum l_\mathrm{i}$	sum of the measured length of each individual intercept

l_x	arithmetic mean linear intercept in phase x, in micrometres (μm)
N	number of grain boundaries traversed in or between specified phases
n	number of WC, carbonitride or cubic carbide grains intercepted
m	magnification
$m_{\max}$	maximum magnification
$m_{\min}$	minimum magnification

5 Principle

This part of ISO 4499 addresses the issue of good practice for the measurement of a mean value for the hard phase and binder phase size in hardmetals other than straight WC/Co. It recommends the use of a linear intercept technique for obtaining data on feature sizes. The measurements are to be made using good practice for the preparation of suitable microstructures for examination outlined in ISO 4499-1.

Methods of metallographic preparation and etching techniques are as important as the phase size measurement method (see also ASTM B 657, ASTM B 665, Reference [1] and Reference [2]). Basic methods are described in ISO 4499-1. Further relevant information is given in Clause 8. The principal types of hardmetal considered are those that contain cubic carbides as well as WC and those that are based on TiC or Ti(C,N).[3][4][5] A cubic carbide phase is defined as a carbide having a cubic lattice, such as, for example, TiC or TaC, and which usually also contains W in solid solution after sintering. These materials typically contain hard phases that have grains with a core/rim structure. Guidelines to measure these internal details are included in ISO 4499–2:2008, Annex A.

The most direct way to measure the phase size is to polish and etch a cross-section of the microstructure and then to use quantitative metallographic techniques to measure a mean value for the feature size, either by area counting or by linear intercept techniques.

The following are three ways by which the mean size by number of the various phases can be defined:

- by length (of a line across a 2D section of a phase);
- by area (of 2D sections of phase regions);
- by volume (of individual phase regions).

A number average is obtained by counting each measurement of the parameter of interest (length, area or volume) and dividing the total value of the parameter (length, area or volume) by the number of this parameter counted.

The values for phase size most used to date have been based on a length parameter. This can be obtained in the following several ways, for example:

- by parallel lines or circles as described in ASTM E112;
- by linear intercept, called the Heyn method, from a straight line drawn across the structure;
- by equivalent circle diameter (see ISO 4499-2), this is obtained by measuring hard phase grain areas and then taking the diameter of a circle of equivalent area.

6 Apparatus

6.1 Metallographic optical microscope, or other suitable equipment permitting observations and measurements on a screen up to the required magnification.